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(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
2 May 2002 (02.05.2002)

PCT

(10) International Publication Number
WO 02/35265 A2

(51) International Patent Classification⁷: **G02B 6/00**

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(21) International Application Number: PCT/US01/42290

(22) International Filing Date:
25 September 2001 (25.09.2001)

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/693,445 20 October 2000 (20.10.2000) US

(84) Designated States (*regional*): European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR).

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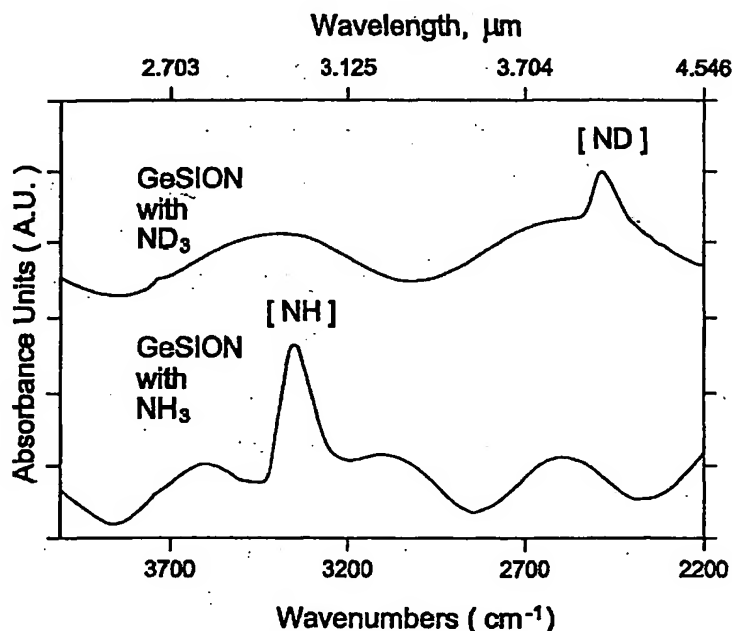
Published:

— without international search report and to be republished
upon receipt of that report

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: USING DEUTERATED SOURCE GASES TO FABRICATE LOW LOSS GeS₁₀ SiON WAVEGUIDES



(57) Abstract: The present invention provides a method of manufacturing optical devices which includes the steps of providing a substrate and forming at least one optical layer on the substrate. The optical layer is formed by a CVD process which includes a deuterated source gas. The present invention also provides an optical device which includes a substrate and an optical layer including deuterium.

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USING DEUTERATED SOURCE GASES TO FABRICATE LOW LOSS GeSiON SiON WAVEGUIDES

BACKGROUND OF THE INVENTION

1. Field of Invention

5 The present invention is directed generally to the manufacture of optical waveguides and more particularly to the use of deuterated source gases to manufacture optical waveguides.

2. Description of the Related Art

Practical optical devices must be fabricated so as to direct the light
10 energy. Commonly, this is achieved by creating a waveguide. In the waveguide, a cladding layer of lower refractive index (typically 1.44) directs light by internal reflectance to an optical core of higher refractive index (typically 1.45-1.5). Both the core and cladding layer can be made from many different materials. Common materials
15 include glasses of $\text{SiO}_2\text{-GeO}_2$, $\text{SiO}_2\text{-B}_2\text{O}_3\text{-P}_2\text{O}_5$, $\text{SiO}_2\text{-GeO}_2\text{-B}_2\text{O}_3\text{-P}_2\text{O}_5$, SiO_2 and SiON. Silicon dioxide, silicon nitride and silicon oxynitride are materials which are particularly valued for their optical properties, in particular their high optical transparency and wide range of refractive indices (1.45-2.5). These materials are used
20 in a host of optical devices. The devices include, for example, planar waveguides, arrayed waveguides (AWG), wavelength demultiplexers, power splitters, optical couplers, phasers, and variable optical attenuators (VOA).

Typically, chemical vapor deposition (CVD) is used to deposit layers of silicon dioxide, silicon nitride or silicon oxynitride. In the CVD process, the substrate is placed on a heated susceptor in a quartz reaction chamber and then the reactant gases are introduced into the chamber. Typically, the gases react on the surface of the substrate and form a deposited layer. However, some reactions may also occur as the gases flow into the chamber. The most common gases for the deposition of silicon dioxide, silicon nitride and silicon oxynitride are silane (SiH_4), chlorinated silane ($\text{SiH}_x\text{Cl}_{4-x}$), nitrous oxide (N_2O), ammonia (NH_3) and nitrogen (N_2). These gases are inexpensive and can be purchased in great abundance.

Although the CVD process is the preferred process for depositing many of the materials used to manufacture optical devices, it is not without problems. The use of ammonia and silane in the production of silicon nitride and silicon oxynitride results in the incorporation of large amounts of hydrogen (up to 20 at% for silicon nitride) in the optical film.

The incorporated hydrogen generates significant optical losses at the 1550 nm optical communication band due to a strong overtone of the N-H bond. Figure 1 illustrates the loss spectrum of conventionally processed silicon oxynitride, i.e., the loss over a range of wavelengths. The peak in loss is due to N-H absorption. In conventional manufacturing processes, the silicon oxynitride contains a significant amount of hydrogen. The figure clearly illustrates the deleterious effect of the overtone of the N-H bond. The center of the loss peak occurs at a wavelength of approximately 1510 nm. This is just 40 nm from 1550 nm, a preferred optical communications wavelength.

It is possible to remove much of the entrapped hydrogen with high temperature thermal annealing. However, the optical SiON film can blister and crack at the high temperature, rendering the device useless.

Therefore, it would be desirable to develop a method to manufacture optical devices which did not result in the incorporation of hydrogen in the optical SiON film and high losses at 1550 nm. Furthermore, it would be desirable to develop a process having the benefits of the speed and control of the conventional CVD process without resorting to a high temperature anneal to drive out the hydrogen.

SUMMARY OF THE INVENTION

The present invention provides a method of manufacturing optical devices comprising providing a substrate and forming at least one optical layer on the substrate by a CVD process including at least one deuterated source gas.

5 The present invention also provides an optical device comprising a substrate and an optical layer including deuterium.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a plot of the loss spectrum of silicon oxynitride in the vicinity of the N-H absorption peak.

10 Figure 2 is a plot of the FTIR spectra of GeSiON films deposited with ND₃ and with NH₃.

Figure 3 is a cross section of an embodiment of the invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention is directed to reducing the optical transmission loss in a waveguide by reducing the hydrogen content in the waveguide. Figure 3 shows the cross section of a planar waveguide manufactured according to a preferred embodiment of the present invention. In this embodiment, an insulating buffer layer 102 is deposited on a substrate 101. A waveguide core 103 including deuterium is then deposited on the buffer layer 102 and the entire structure is coated with a cladding layer 104. As demonstrated below, the use of deuterated source gases is effective in reducing the hydrogen content of the waveguide.

Silicon is the preferred material for the substrate 101. However, the substrate 101 may be made out of any material suitable for supporting the waveguide core 103. Example substrate materials include, but are not limited to, GaAs, InP, SiO₂, Si₃N₄, ceramics and plastics.

25 The preferred material for the buffer layer 102 is silicon oxynitride (SiON) or germanium doped silicon oxynitride (GeSiON). More preferably, the material for the buffer layer 102 is deuterated silicon oxynitride (SiON) or deuterated

germanium doped silicon oxynitride (GeSiON). However, any suitable material can be used. For optimum results, the buffer layer 102 should have an index of refraction less than the index of refraction of the waveguide core 103. The buffer layer 102 may be omitted if the substrate is formed from a suitable material with a lower index of refraction than the core.

The preferred material for the cladding layer 104 is SiON or GeSiON. More preferably, the preferred material for the cladding layer 104 is deuterated SiON or deuterated GeSiON. However, any suitable material, such as plastics for example, can be used. For optimum results, the cladding layer 104 should have an index of refraction less than the index of refraction of the waveguide core 103.

The core 103 of the optical waveguide preferably comprises deuterated germanium doped silicon oxynitride ($\text{Ge}_w\text{Si}_x\text{O}_y\text{N}_z$), where the sum of w , x , y and z is equal to 1. More preferably, the core 103 comprises deuterated silicon oxynitride ($\text{Si}_x\text{O}_y\text{N}_z$), where the sum of x , y and z is equal to 1. The deuterium replaces hydrogen and thereby reduces the hydrogen content in the waveguide. The index of refraction of the core is preferably between 1.44 and 2.2. More preferably, the index of refraction of the core is between 1.6 and 1.8. Furthermore, transmission losses due to attenuation are preferably less than 4.0 dB/cm in multimode slab waveguides and less than 2.0 dB/cm in single mode slab waveguides. More preferably, the transmission losses due to attenuation are less than 1.5 dB/cm in multimode slab waveguides and less than 0.2 dB/cm in single mode slab waveguides at 1550 nm.

By using deuterium source gases in manufacturing the core 103, the hydrogen content of the core 103 is reduced and consequently the optical loss is reduced. This is shown, for example, by the Fourier transform infrared (FTIR) spectra of germanium doped silicon oxynitride (GeSiON) films are illustrated in Figure 2.

A film deposited with NH_3 is represented by the bottom spectrum in Figure 2. This spectrum shows, like Figure 1, that the NH stretch is at 3310 cm^{-1} (3.02 μm), which places the overtone absorption peak approximately at 1510 nm, which is near the communications wavelength. In contrast, the top spectrum in figure 2 represents a the germanium doped silicon oxynitride film formed with deuterated ammonia (ND_3) instead of regular ammonia (NH_3). As shown, the absorption peak is

shifted from 3.02 μm to 4.08 μm . The overtone peak therefore shifts to 2004 nm, far from 1550 nm, the optical communications wavelength, by substituting ND_3 for the NH_3 source gas during deposition. The source gas may be partially deuterated.

The use of deuterated silane, deuterated disilane and deuterated germane produce similar results. These source gases may also be partially deuterated. The use of any of these gases alone or in combination is beneficial because losses at 2004 nm due to the N-D bond are not significant for communications at 1550 nm.

The use of the deuterated core material has been described and illustrated by way of an optical waveguide. However, this is but one device which can be fabricated according to the present invention. Other devices which may also benefit from the material of the present invention include, but are not limited to, an optical waveguide, an arrayed waveguide, a wavelength demultiplexer, a power splitter, an optical coupler, a phaser, and a variable optical attenuator.

The core of the optical waveguide is preferably deposited by chemical vapor deposition (CVD). Low pressure CVD (LPCVD), atmospheric pressure CVD (APCVD) and plasma assisted CVD (PECVD) can be used. However, PECVD is the preferred method. One example of PECVD deposition is described below.

EXAMPLES

Deuterated silicon oxynitride and deuterated germanium doped silicon oxynitride films were deposited with an STS Multiflex PECVD system. This system is a parallel plate reactor where the precursor gases enter through an array of holes in the top electrode (showerhead) and the sample rests on the bottom electrode. The bottom electrode is a non-rotating heated platen. The reaction gases included silane (SiH_4), germane (GeH_4), nitrous oxide (N_2O), deuterated ammonia (ND_3) and nitrogen (N_2). Regular ammonia (NH_3) was also available for making the comparative examples. The refractive index, optical propagation loss and film thickness were determined with a prism coupling system.

An initial series of thin germanium doped silicon oxynitride films were deposited with and without deuterated ammonia to evaluate the reduction in waveguide loss. These films were deposited on Si(100), SiO_2 and Corning 1737 glass substrates to

form multimode slab waveguides. The deposition parameters for these films are in Table 1.

TABLE 1

Recipe ID	EX. A	EX. B	EX. C	EX. D	EX. E	EX. F
Rf (380kHz) Power (W)	400	400	400	400	400	400
Pressure (mtorr)	500	400	400	400	500	400
Substrate Temperature (C)	350	350	350	350	350	350
Showerhead Temperature (C)	225	225	225	225	225	225
5% SiH ₄ /Ar (sccm)	200	100	100	200	200	100
2% GeH ₄ /Ar (sccm)	250	400	400	250	250	400
N ₂ O (sccm)	200	117	117	200	200	117
ND ₃ (sccm)	200	50	100	300	0	0
NH ₃ (sccm)	0	0	0	0	200	50
N ₂ (Sccm)	1600	2000	2000	1600	1600	2000
Deposition Time (min)	40	40	40	40	40	40

- 5 Films from the initial round of GeSiON films with ND₃ were smooth and uniform with a slight green coloration as deposited. The film thicknesses (t, μm) and refractive indexes (n) were measured by prism coupling at two wavelengths, 652 nm and 1550 nm. The prism coupling measurements demonstrate that the thickness and indexes are very near that of films deposited with NH₃. Table 2 summarizes the
- 10 thickness and refractive index measurements.

TABLE 2

	n (632 nm)	t (632), μm	n (1550 nm)	t (1550), μm	t (ave), μm
EX. A	1.6732	3.6012	1.6482	3.5616	3.5814
EX. B	1.7398	3.3384	1.7105	3.3607	3.3496
EX. C	1.8308	3.2623	1.7897	3.2462	3.2543
EX. D	1.7073	3.2744	1.6819	3.1631	3.2188

Table 3 is a comparison between attenuation measurements on multimode slab germanium doped silicon oxynitride waveguides deposited with ND_3 (Examples A to D) and multimode slab germanium doped silicon oxynitride waveguides deposited with NH_3 (Examples E and F).

TABLE 3

Sample	Loss in dB/cm
EX. A	2.5
EX. B	2.1
EX. C	3.5
EX. D	1.3
EX. E	8
EX. F	11

The optical propagation loss in multimode slab waveguides ranged from 1.3 to 3.5 dB/cm for films deposited with ND_3 . By comparison, the propagation loss in comparable films deposited with NH_3 ranged from 8-11 dB/cm.

Table 4 summarizes and compares the compositions of the GeSiON films deposited with ND_3 with those deposited with NH_3 .

TABLE 4

Atomic Fraction							
Sample		Si	Ge	O	N	H	D
EX. E	NH ₃	0.184	0.087	0.295	0.250	0.184	-
EX. A	ND ₃	0.153	0.104	0.317	0.190	0.086	0.150
EX. F	NH ₃	0.121	0.196	0.425	0.149	0.109	-
EX. A	ND ₃	0.093	0.228	0.377	0.122	0.090	0.090

The samples prepared with ND₃ have significantly less hydrogen incorporation than the samples prepared with NH₃. Samples deposited with ND₃ show lower nitrogen levels. This is believed to result from a lower ND₃ flow than NH₃ because the flow controllers were not re-normalized.

A second series of thin germanium doped silicon oxynitride films were deposited with deuterated ammonia to evaluate the reduction in waveguide loss. In this series, two samples were deposited with a waveguide core over a 13-15 μ m cladding layer on a silicon wafer to form a single mode waveguide. The deposition parameters are summarized in Table 5.

TABLE 5

Recipe ID	G		H		I		J	
	Under-clad	Core	Under-clad	Core	Under-clad	Core	Under-clad	Core
Rf (380kHz) Power (W)	400	400	400	400	400	400	400	400
Pressure (mtorr)	400	400	400	400	400	400	400	400
Substrate Temperature (C)	350	350	350	350	350	350	350	350
Showerhead Temperature (C)	225	225	225	225	225	225	225	225
5% SiH ₄ /Ar (sccm)	100	100	100	100	100	100	100	100
2% GeH ₄ /Ar (sccm)	400	400	400	400	400	400	400	400
N ₂ O (sccm)	125	117	125	117	125	117	125	117
ND ₃ (sccm)	50	50	50	50	0	0	0	0
NH ₃ (sccm)	0	0	0	0	50	50	50	50
N ₂ (Sccm)	2000	2000	2000	2000	2000	2000	2000	2000
Deposition Time (min)	135	40	160	60	135	40	160	60

As in the initial series of films, the film thicknesses and refractive indexes were measured by prism coupling at two wavelengths, 652 nm and 1550 nm. The prism coupling measurements demonstrate that the thickness and indexes are very near that of films deposited with NH_3 . Table 6 summarizes the thickness and refractive index measurements.

TABLE 6

		n (632 nm)	t (632), μm
EX. G	Clad	1.6875	13.4047
	Core	1.6997	6.5692
EX. H	Clad	1.6916	16.0999
	Core	1.7007	6.6219

Table 7 is a comparison between attenuation measurements on single mode slab germanium doped silicon oxynitride waveguides deposited with ND_3 (Examples G and H) and single mode slab germanium doped silicon oxynitride waveguides deposited with NH_3 (Examples I and J).

TABLE 7

Sample	loss in dB/cm
EX. G	1.3
EX. H	2.0
EX. I	7
EX. J	7

The optical propagation loss in single mode waveguides ranged from 1.3 to 2.0 dB/cm for films deposited with ND_3 . By comparison, the propagation loss in comparable films deposited with NH_3 was 7 dB/cm.

A third series of deuterated thin film single mode waveguides were manufactured to study the effect of various GeSiON/SiON core/cladding configurations. In this series, three combinations were tested. These include, GeSiON

core/clad, SiON core/clad and SiON core on GeSiON clad. The deposition parameters are summarized in Table 8.

TABLE 8

	K		L		M		N	
Recipe ID	Under-clad	Core	Under-clad	Core	Under-clad	Core	Under-clad	Core
Rf (380kHz) Power (W)	400	400	400	400	400	400	400	400
Pressure (mtorr)	350	350	350	350	350	350	350	350
Substrate Temperature (C)	350	350	350	350	350	350	350	350
Showerhead Temperature (C)	225	225	225	225	225	225	225	225
5% SiH ₄ /Ar (sccm)	100	100	260	260	100	260	100	0
2% GeH ₄ /Ar (sccm)	400	400	0	0	400	0	400	0
5% SiD ₄ /Ar (sccm)	0	0	0	0	0	0	0	260
N ₂ O (sccm)	125	120	35	35	126	35	126	33
ND ₃ (sccm)	45	45	60	60	45	75	45	100
N ₂ (Sccm)	3500	3500	3500	3500	3500	3500	3500	3500
Deposition Time (min)	160	60	205	55	165	57	165	90

5 In the third series, both the propagation loss and the wafer warpage was measured. The results of these experiments are summarized in Table 9.

TABLE 9

Sample	Loss in dB/cm	Wafer warpage TIR (μm)
EX. K	1.12	20.5
EX. L	<0.2	217.8
EX. M	0.38	8.0
EX. N	<0.2	121

10 The first sample (Example K), a GeSiON core on a GeSiON cladding layer, exhibits approximately a 1 dB/cm propagation loss with low wafer warpage. The second sample (Example L), a SiON core on a SiON cladding layer, had a propagation loss near the 0.2 dB/cm detection limit of the test equipment. However, the wafer

warpage is very high. Depositing a SiON core on a GeSiON cladding, the third sample (Example M), resulted in a slightly higher loss of 0.38 dB/cm while reducing wafer warpage. Depositing a SiON core on a GeSiON cladding using deuterated ammonia and deuterated silane, the fourth sample (Example N), resulted in propagation losses
5 below 0.2 dB/cm. However, wafer warpage increased significantly.

The foregoing description of the invention has been presented for purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise form disclosed, and modifications and variations are possible in light of the above teachings or may be acquired from practice of the
10 invention. The drawings and description were chosen in order to explain the principles of the invention and its practical application. It is intended that the scope of the invention be defined by the claims appended hereto, and their equivalents.

WE CLAIM:

1. A method of manufacturing optical devices comprising:
providing a substrate; and
forming at least one optical layer on said substrate by a CVD process
5 including at least one deuterated source gas.
2. The method of claim 1, wherein the deuterated source gas is selected from the group consisting of deuterated ammonia, deuterated silane, deuterated disilane and deuterated germane.
3. The method of claim 2, wherein the source gas is partially
10 deuterated.
4. The method of claim 1, wherein the CVD process is selected from the group consisting of APCVD, LPCVD and PECVD.
5. The method of claim 1, wherein said step of forming at least one optical layer includes forming an optical layer with a wavelength of an overtone about
15 2004 nm.
6. The method of claim 1, wherein said step of forming at least one optical layer includes forming an optical layer with an index of refraction between 1.45 and 2.2.
7. The method of claim 6, wherein said step of forming at least one
20 optical layer includes forming an optical layer with an index of refraction between 1.6 and 1.8.
8. The method of claim 1, wherein said step of forming at least one optical layer includes forming a layer of silicon oxynitride.
9. The method of claim 1, wherein said step of forming at least one
25 optical layer includes forming a layer of germanium doped silicon oxynitride.
10. The method of claim 1, wherein said optical layer exhibits propagation losses below 4 dB/cm.
11. The method of claim 1, wherein said optical layer exhibits propagation losses below 0.2 dB/cm.
- 30 12. The method of claim 8, wherein said optical layer exhibits propagation losses below 4 dB/cm.

13. The method of claim 8, wherein said optical layer exhibits propagation losses below 0.2 dB/cm.
14. The method of claim 8, further comprising:
forming a cladding layer.
- 5 15. The method of claim 14, wherein said cladding layer is either deuterated silicon oxynitride or deuterated germanium doped silicon oxynitride.
16. The method of claim 14, further comprising:
forming a buffer layer.
17. The method of claim 16, wherein said buffer layer is either
10 deuterated silicon oxynitride or deuterated germanium doped silicon oxynitride.
18. An optical device comprising:
a substrate; and
an optical layer comprising deuterium.
19. The optical device of claim 18, wherein the optical layer
15 comprises deuterated silicon oxynitride.
20. The optical device of claim 18, wherein the optical layer
comprises deuterated silicon oxynitride or deuterated germanium doped silicon oxynitride.
21. The optical device of claim 18, wherein the device further
20 comprises a cladding layer.
22. The optical device of claim 21, wherein the cladding layer
comprises deuterated silicon oxynitride or deuterated germanium doped silicon oxynitride.
23. The optical device of claim 22, wherein the device further
25 comprises a buffer layer.
24. The optical device of claim 23, wherein the buffer layer
comprises deuterated silicon oxynitride or deuterated germanium doped silicon oxynitride.
25. The optical device of claim 18, wherein the wavelength of an
30 overtone in the patterned optical layer is about 2004 nm.

26. The optical device of claim 18, wherein said optical layer exhibits propagation losses below 4 dB/cm.

27. The optical device of claim 18, wherein said optical layer exhibits propagation losses below 0.2 dB/cm.

5 28. The optical device of claim 22, wherein said optical layer exhibits propagation losses below 4 dB/cm.

29. The optical device of claim 22, wherein said optical layer exhibits propagation losses below 0.2 dB/cm.

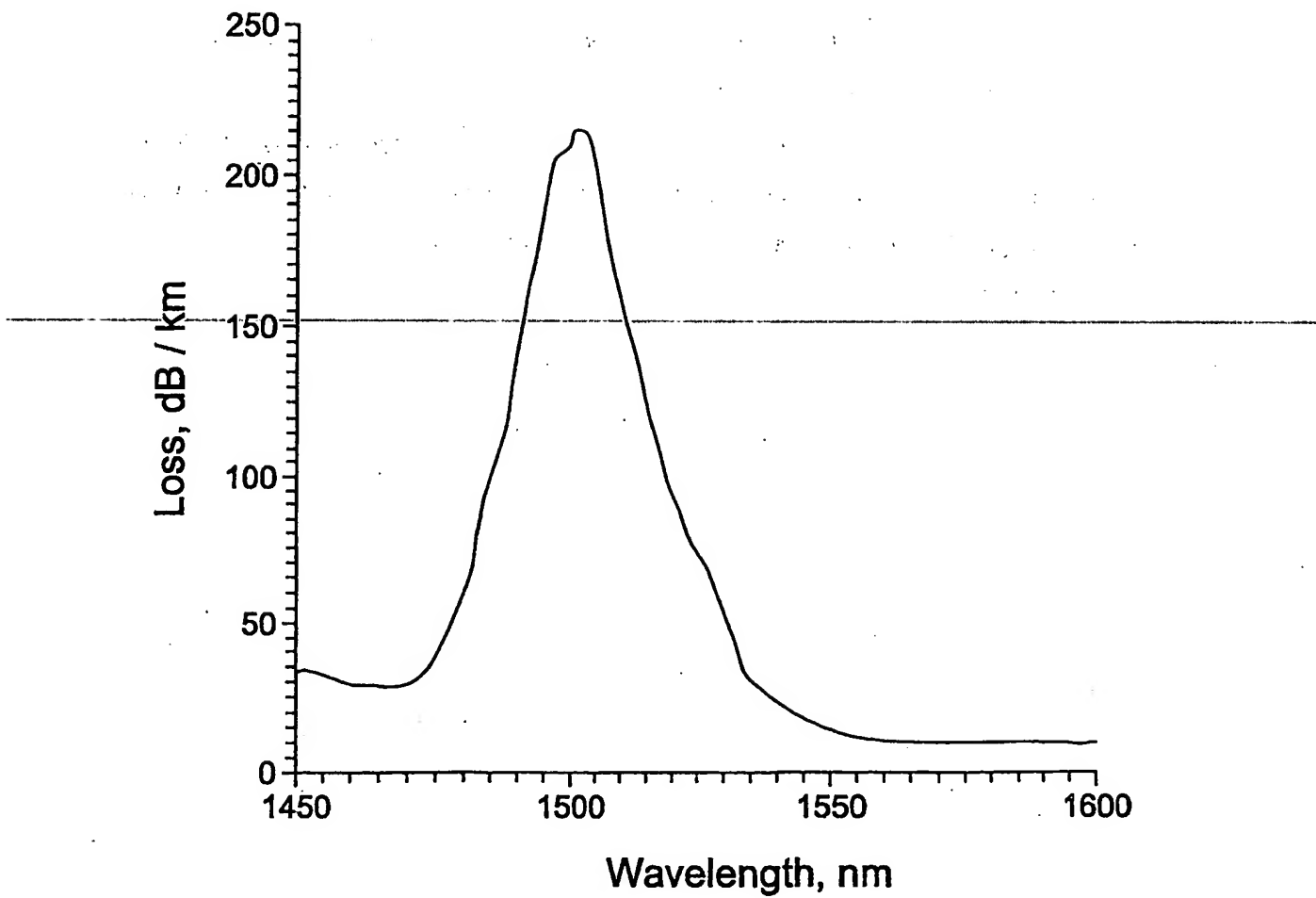
10 30. The optical device of claim 22, wherein said optical layer exhibits an index of refraction between 1.45 and 2.2.

31. The optical device of claim 22, wherein said optical layer exhibits an index of refraction between 1.6 and 1.8.

15 32. The optical device of claim 18, wherein said optical device is selected from the group consisting of an optical waveguide, an arrayed waveguide, a wavelength demultiplexer, a power splitter, an optical coupler, a phaser, and a variable optical attenuator.

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FIG.1



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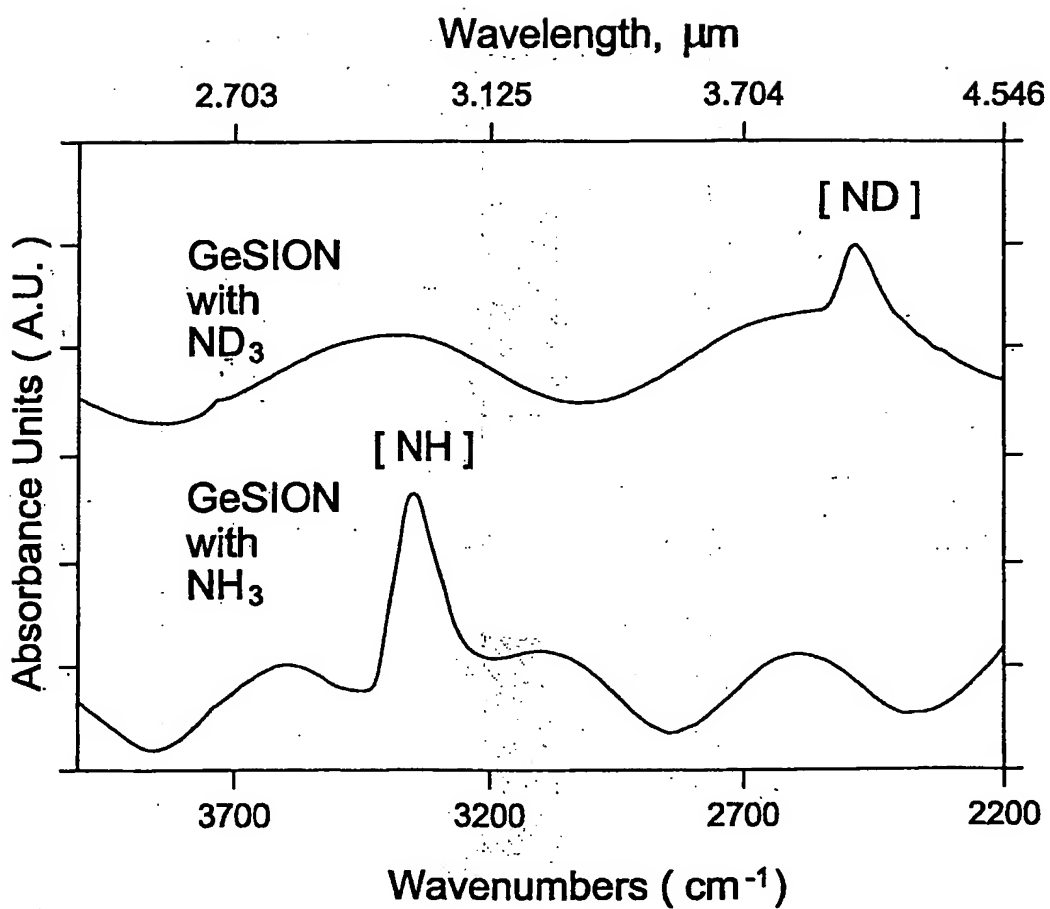
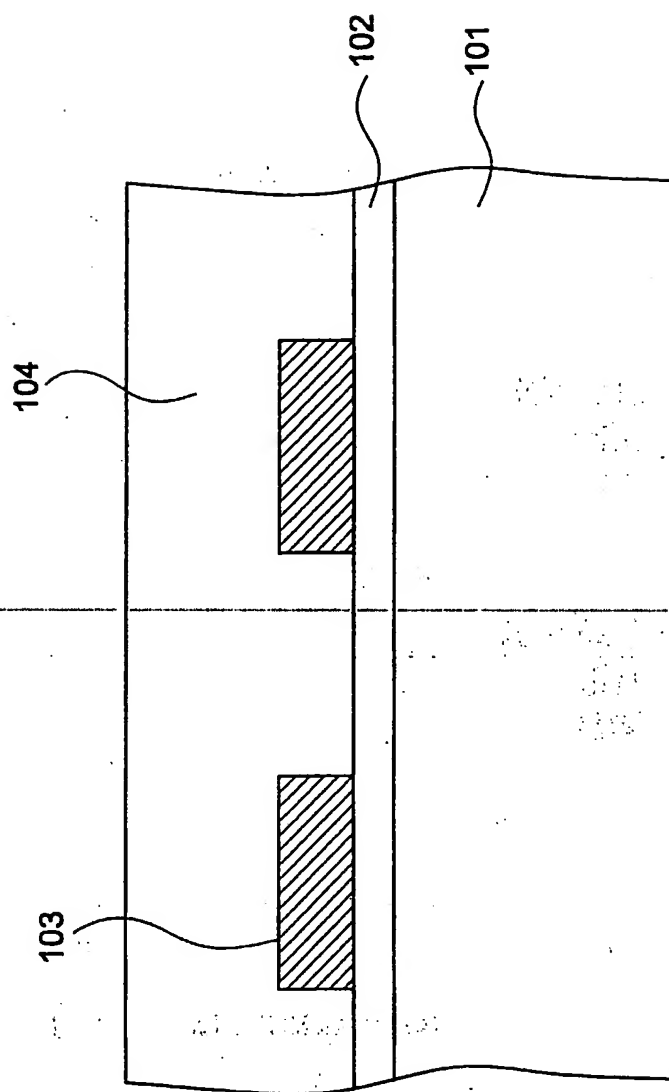


FIG.2

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FIG. 3



(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
2 May 2002 (02.05.2002)

PCT

(10) International Publication Number
WO 02/035265 A3

(51) International Patent Classification⁷: G02B 6/132

(21) International Application Number: PCT/US01/42290

(22) International Filing Date:
25 September 2001 (25.09.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/693,445 20 October 2000 (20.10.2000) US

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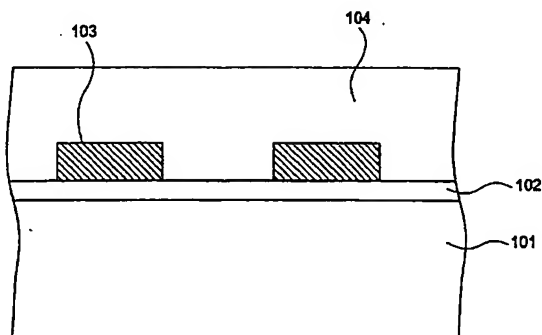
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Patent Department, SP TI 3-1, Corning, NY 14831 (US).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU,
AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU,
CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
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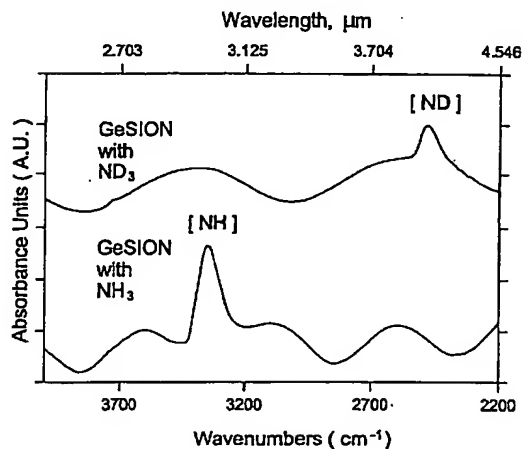
(84) Designated States (*regional*): European patent (AT, BE,
CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC,
NL, PT, SE, TR).

[Continued on next page]

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WO 02/035265 A3



Published:

— with international search report

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(88) Date of publication of the international search report:

23 January 2003

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/42290

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 602B6/132

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 602B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, INSPEC, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO 01 64594 A (WIESMANN DOROTHEA W ;BLOECHL PETER (CH); HORST FOLKERT (CH); BEYEL) 7 September 2001 (2001-09-07) page 7 -page 10	1-32
X	PATENT ABSTRACTS OF JAPAN vol. 1997, no. 07, 31 July 1997 (1997-07-31) -& JP 09 078244 A (CANON INC), 25 March 1997 (1997-03-25) abstract	1-4, 15
Y	EP 0 673 895 A (AT & T CORP) 27 September 1995 (1995-09-27) page 1 -page 2	1-32
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

26 September 2002

Date of mailing of the international search report

08/10/2002

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INTERNATIONAL SEARCH REPORT

International Application No

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GERMANN R ET AL: "SILICON-OXYNITRIDE LAYERS FOR OPTICAL WAVEGUIDE APPLICATIONS" ELECTROCHEMICAL SOCIETY PROCEEDINGS, ELECTROCHEMICAL SOCIETY, PENNINGTON, NJ, US, vol. 99-6, 2 May 1999 (1999-05-02), pages 169-181, XP000987166 ISSN: 0161-6374 page 1 -page 2	1-32
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 01/42290

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